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Process and system for measuring the composition of materials

The object of the invention is a measurement process by which the composition of materials can be determined with greater ease. Due to said determination, the process can be utilized to good advantage in process control for purposes of display, control, and recording. The process is especially advantageous when used in the chemical, food, and pharmaceutical industries. A suitable system for implementation of the process is a further object of the invention.

With the progress of new technologies, high-quality products can be manufactured only using automatic controls. Automation of progressing technologies (in the food, chemical, and pharmaceutical industries, etc.) demands that, during the manufacturing process, information be as complete as possible regarding the materials involved in each phase of the work (raw materials, semi-finished products, finished products) and their composition, quality, and characteristics.

Product quality can be unequivocally described by identifying components in percent by weight, in other words in hundredths or weight fractions, so that measurements of quality can be derived from measurements of composition. The inventors feel justified in making the preceding statement because the composition of any given substance given in terms of percent by weight, when reported with sufficient accuracy, is an unequivocal determinant of all other characteristics (commonly considered to fall under the rubric of quality) pertaining to the product. Thus, the composition of a substance determines all of that substance's measurable physical attributes. Yet when only one physical attribute capable of being measured well is known, any given characteristic of the substance's makeup can be determined only if such physical attribute depends solely on the compositional characteristic in question, i.e. if it is selective solely in relation to that compositional value. However, a sufficient number of selective physical attributes can be found either with difficulty, or by a method of measurement that is cumbersome or imprecise, or possibly by a measuring device (such as a mass spectrograph, gas chromatograph, etc.) that is very costly.

The measurement process according to the invention is based on the realization that a specific relationship exists between the compositional characteristics and the physical attributes. If, therefore, of all the physical attributes, a suitable number of attributes that can be measured quickly, inexpensively, and accurately can be selected, such selected group of attributes will be unequivocally determined by the compositional characteristics. However complicated the relationship may be, or if the particular physical attributes do not depend solely on one compositional characteristic but on all of them, the physical attributes are not necessarily selective.

The measurement process is essentially as follows:
A study is carried out to assess the physical attributes that can, for the substance in question whose makeup is to be determined, be ascertained quickly and accurately using simple instruments. Next, based on measurements of those physical attributes taken from samples of the

same substance whose makeup is known, the relationship between the compositional characteristics and the physical attributes is found. Said relationship is ascertained especially in proximity to the desired composition. With process control, the composition of the particular products varies considerably in close proximity to the work site, and for that reason it is permissible to linearize the relationships in the vicinity of the makeup at the work site.

The linear relationships between the compositional characteristics and the physical attributes are inverted. If the samples of known composition are selected accordingly, the inversion can be performed in each and every case, thereby providing linear relationships that can be used, given known physical attributes, to determine the compositional characteristics. The relationships are valid over a broader range for a given substance, but a high degree of accuracy is attainable only in a certain proximity to the work site. Accordingly, after measuring the physical attributes of a substance whose makeup is not known, its composition can be determined with the aid of the transformational relationships. The process is not suitable for universal determinations of composition, and applies only to substances where the physical attributes are available for samples of known composition.

The transformation of the physical attributes into compositional characteristics using the composition measurement device according to the invention may also be carried out automatically. The system should have input and output signal capabilities corresponding to the physical attributes and compositional characteristics, respectively, between which the characteristic transformational relationships for the substance that is to undergo examination are valid. The system can be electronic when the measured values of the physical parameters are available in the form of incoming current emitted by a remote transmitter. In such cases, the compositional characteristics are obtained as the sum of voltages generated accordingly. A system operating on the basis of any physical principle (e.g. mechanical, pneumatic, etc.) is suitable for implementation of the process according to the invention. It is essential that the system connect the input and output signals with the aid of the appropriate system of linear algebraic equations.

Aside from the compositional characteristics, other additional characteristics (e.g. temperature, pressure, relative moisture content, etc.) will have a disturbing effect on the measured physical attributes. However, even the effect of these disturbing variables can be accounted for using linear relationships, thereby allowing compensation for the disturbance to the substance of unknown composition by measuring the disturbing variable. Due to linearization, a method of adjustment can be incorporated in the transformational system, thereby achieving, for example, an automatic, complex, adjusted measurement.

The process according to the invention is not only suitable for the measurement of composition. The process can be used to measure an arbitrary group of characteristics pertaining to a material system by measuring another group of characteristics.

As can be seen from the preceding discussion, this fundamentally novel process and this fundamentally novel system surpass former systems having similar objectives with respect to accuracy, reliability, simplicity, manufacturing costs, etc. and, thus, they represent a qualitative technical advance.

The process according to the invention is explained below in terms of several examples and drawings.

Example 1. Continuous production of a liqueur.

The object is to produce a solution by mixing and homogenization, which is essentially a mixture containing three components in a prescribed ratio. One component is water, another is sugar, and the third is some type of alcohol containing aromatic and coloring agents. According to Fig. 1, the manufacturing process is as follows: The water, sugar solution, and alcohol containing the coloring agents are stored in three containers 1, 2, and 3. The three "basic ingredients" pass through pipes 6 [sic, labeled 7 in drawing], 8, and 9 equipped with valves (organs for adding to the mixture) 4, 5, and 6 into the mixing and homogenization vessel 10, from

where the final mixture flows in the direction indicated by the arrow 11 to the filling machine. The three basic ingredients are dosed in the proper ratio by adjusting the valve lift. In the case of fractionated production, the "prescribed composition" of the mixture could only be checked by chemical methods. Because the chemical process was so slow (taking more than one day to yield the results of the analysis), it could not be used as the basis for automatic control. However, automatic control is absolutely necessary in the case of continuous production. It is possible to determine the percent of the components by weight, i.e. the compositional characteristics, quickly and accurately with the aid of the process according to the invention, namely an alteration process. In order to determine the three ingredients, two physical parameters must be measured. The instrument transforms the physical attributes into the compositional characteristics that are being sought. It provides a continuous signal derived from the compositional characteristics, and that signal thus provides the basis for implementation of the automatic control. Fig. 2, by way of example, shows the circuitry of the "transformational instrument" used in implementing the automatic process control according to Fig. 1 with the aid of the measurement alteration process.

According to Fig. 1, the specific gravity μ and the refraction index ν of the liqueur are measured immediately after the mixing and homogenization vessel 10. The measurements of the specific weight and refraction index are transformed by measurement amplifiers (remote transmitters) 12 and 13 into an electric current of $i_1(\mu)$ and $i_2(\nu)$, respectively, proportionally equivalent to the value of the specific weight μ and the value of the refraction index ν, e.g. into a VHF signal from 0 to 5 mA. Using algebraic operations, the "transformational instrument" 14 converts the two current signals sent as input and will emit as output the variables of sugar weight percent S_c and alcohol weight percent S_a in the form of voltage signals. These signals are then used, with the aid of the commercially available control units R_1 and R_2 and execution mechanisms 15 and 16, to adjust the valves 5 and 6 that change the concentration of the sugar and alcohol, respectively. Production output is regulated by means of the lift of valve 4 located in line 7 of the water container 1.

Fig. 2 shows the circuitry of the "transformational instrument" 14 according to Fig. 1. The magnitude of the two physical attributes μ and ν depends on both sugar and alcohol content, and vice versa, the magnitude of both physical attributes plays a role in the determination of the compositional characteristics. This relationship is described by the following equations (linear approximation):

$S_a = A\mu + B\nu + C$ (for alcohol)

$S_c = D\mu + E\nu + F$ (for sugar),

Where A, B, C, D, E, and F are transformational matrix elements, each having a constant value for one substance.

In other words, the instrument completes the transformation (mapping) according to the equations. Current i_1, which is proportionally equivalent to the specific gravity μ, will flow through a resistance R_A corresponding to the value A, causing a decrease in voltage that is proportionally equal to the product $A\mu$. The voltage that is proportionally equal to the product $B\nu$ is wired in series to it, and decreases through the resistance R_B that is proportionally equal to the value B when acted upon by the current i_2 that is proportionally equal to the refraction index ν. Finally, the decreasing voltage joins the voltage last mentioned above through the resistance R_C that corresponds to the last member of the first equation of the constant C. In the circuitry, the signs of the coefficients appearing in the equations can also be taken into account. In the case of liqueur, for example, the coefficient A is negative. The equation takes this fact into account by the opposing series circuit of the falling voltage through the resistance R_A.

Example 2. Continuous butter production.

In the continuous production of butter, churning or thermal treatment is used to prepare an emulsion in which care must be taken to maintain the prescribed weight percent of the three components essentially by means of automatic control. One component is butter fat, the second

water (buttermilk), and the third air. Automatic control is possible only if information regarding the weight percent values of all three components is available at all times. Both the recourse to the method of measurement alteration for construction of the automatic control and the circuitry of the instrument are in every way the same as in the preceding example. In order to determine the three compositional characteristics, two physical attributes must be measured. In the case of butter, one physical attribute is the core absorption μ. It is extremely difficult to determine the specific gravity of substances with a density like that of butter. The second physical attribute is the dielectric constant ε. Although the value of elements in the transformational matrix (A, B, C, D, E, F) differs from that for liqueur, the circuitry of the instrument is identical. Only the values of R_A, R_B, R_C, R_D, R_E, and R_F must be adjusted differently. A further difference from the automatic control used in liqueur production is that when producing butter, the compositional characteristics are not adjusted with the aid of valves but rather by changing the RPM of the churning machine, the temperature of the manufacturing machinery, and the output of the manufacturing machinery.

Example 3. Continuous production of pickling solution.

Pickling solution is the solution needed in order to preserve pickles, peppers, cabbage, etc. The production of pickling solution is very similar to that of liqueur. Here, too, the solution is prepared by mixing and homogenization. The solution itself is a mixture containing several components in a prescribed ratio. The difference is that in this case there are not three but four components. One component is water, another salt, the third sugar, and the fourth vinegar. In order to determine the four components, it is necessary to measure three physical attributes, namely, specific gravity μ, conductivity χ, and refraction index ν. (It is also possible to measure the rotatory power α instead of the refraction index and the pH factor instead of the specific gravity.) When dealing with a pickling solution having four components, the relationship between the compositional characteristics and the physical attributes is described by the following system of equations:

$S_s = A\mu + B\chi + C\nu + D$ (for salt)

$S_c = E\mu + F\chi + G\nu + H$ (for sugar)

$S_e = J\mu + K\chi + L\nu + M$ (for vinegar)

The number of elements in the transformational matrix (the coefficients of the equation system) is 12. Accordingly, twelve resistance values must be set in the "transformational instrument," and it must also be possible to take the sign of those numbers into account. Establishing a relationship between the physical attributes and the current and between the coefficients and the resistance, the compositional characteristics are obtained in the form of voltages.

Example 4. Production of meat paste.

In the production of meat paste, a mass is prepared by mincing, mixing, and homogenization that is essentially a mixture of four components in a prescribed ratio. The four components are protein (meat), fat (lard), salt, and water. The basic ingredients to be dosed are the ground meat containing water and fat, the lard containing water and protein [sic], salt, and water. This is a case where the task of measurement technology can be successfully accomplished by the "measurement alteration" method. In order to determine four components, three physical attributes must be measured, namely: core absorption μ, conductivity χ, and the dielectric constant ε. Instead of the latter value, the relative equilibrium moisture content erp or the core reflection (neutron scatter) can be measured. Instrument circuitry and automatic control design are in all respects the same as those described above.

Let it be noted here that by conserving valuable and useful substances (such as, for example, alcohol, fat, protein, sugar) in the individual production lines with …
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Munich, July 20, 1970

LICENCIA ...

New Patent Claim 1

A process for the determination of groups of technical characteristics that can otherwise be measured only with difficulty or not at all, especially for the determination of compositional characteristics of various chemicals, pharmaceutical products, industrial food products, etc., on the basis of measuring other technical attributes that can be measured easily, quickly, and accurately, **characterized by the fact that** the product whose composition is to be determined is used to prepare an appropriate number of samples with known makeup, or samples with different makeup are obtained by sample collection, whereby the composition parameters of said samples /expressed, for example, as weight fraction, weight percent, volume percent, or other units/ /are determined using conventional methods, and also other types of quickly measurable physical attributes /for example, density, refractive index, optical rotatory power, electrical conductivity, dielectric constant, surface tension, beam attenuation constant, viscosity, etc./ are measured, and the mathematical relationship between the compositional parameters and the physical parameters is established on the basis of these measurements, and said relationship is described in the form of regression equations or in the form of tables, or by realizing a corresponding numerical counting mechanism, or by programming a counting mechanism, and the unknown composition parameters are obtained by measuring the values of the selected physical parameters of the material whose makeup is unknown based on the previously obtained relationships, and should there be deviations from a prescribed target value, the composition is corrected by means of the appropriate control devices.

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[two pages of illustrations]